IS 3228 (1986): Musk Ketone [PCD 18: Natural and Synthetic Fragrance Materials]

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Indian Standard

SPECIFICATION FOR MUSK KETONE

(First Revision)

UDC 665·54 : 547·572

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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April 1987

Indian Standard

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(First Revision)

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(*Continued on page* 12)

Indian Standard

SPECIFICATION FOR MUSK KETONE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 August 1986, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1965. It is being revised with a view to bring it in line with the trade practices in perfumery technology and also to align it with the quality level of the material currently produced and sold in the country.

0.3 The nitro-musks represent an important and widely used group of synthetic perfumery materials which are structurally unrelated to either natural musk of animal origin or musk-like other products of plant origin. However, the odours of the pure crystalline nitro-musks are strongly suggestive, with varying degrees of intensity, of the naturally occurring musk group of perfumery materials.

0.3.1 Of all the nitro-musks, musk ketone ($C_{14}H_{18}N_2O_5$) compares more favourably to the odour of natural musk, being finer, more intense and more animal-like. It has the structural formula given on page 4.

0.4 Musk ketone is not reported to occur in nature. It is prepared by nitration of the corresponding benzene derivative and is purified by recrystallization, employing suitable solvents.

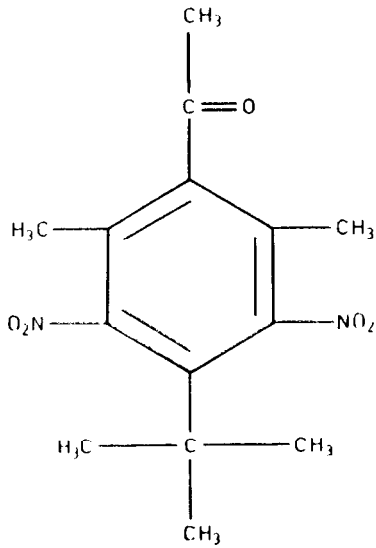
0.5 A new requirement for musk ketone, percent by mass, *Min* along with gas chromatographic method for determination of musk ketone has been included in this revision based on data generated through indigenous testing.

0.6 In the preparation of this standard, considerable assistance has been derived from the following:

EOA No. 25 Standard for nitro-musks (*revised* 1956) and first supplement to the EOA Book of standards and specification (1979). Essential Oil Association of USA, New York.

Technical information bulletin No. 1646. A Boake, Roberts & Co. Ltd, London.

The Givaudan index, 1961. Givaudan-Delawanna, Inc., New York.



[3, 5-dinitro 2, 6-dimethyl 4-tertiary butyl acetophenone (Molecular Mass 294·30)]

MUSK KETONE

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for musk ketone. The material is generally used as a fixative in perfume compositions.

*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 6597-1972* shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall be in the form of very pale yellow platelets or fine crystalline powder.

3.1.2 The material shall also be tested olfactorily and especially for bynotes as prescribed under **4** and **5** of IS : 2284-1963†.

3.2 Solubility — The material shall be soluble, at 25 ± 1°C, in the following solvents in concentrations specified against each:

a) Benzyl benzoate, *Min*	1·4 g/5 ml
b) Diethyl phthalate, *Min*	0·8 g/5 ml
c) Ethyl alcohol (95 percent by volume), *Min*	1·4 g/100 ml

3.3 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR MUSK KETONE

(*Clauses* 3.3 *and* 6.1)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Appendix	Cl No. in IS : 2284-1963*
(1)	(2)	(3)	(4)	(5)
i)	Odour	Closely resembling natural musk	—	4 and 5
ii)	Melting range, °C	135·0 to 136·5	A	—
iii)	Alkali stability	To pass test	B	—
iv)	Musk ketone, percent by mass, *Min*	98	C	—

*Method for olfactory assessment of natural and synthetic perfumery materials.

4. PACKING AND MARKING

4.1 The material shall be supplied in paper-lined tin cans, fibre-board, press-board containers, or in wooden barrels.

*Glossary of terms relating to natural and synthetic perfumery materials.
†Method for olfactory assessment of natural and synthetic perfumery materials.

4.2 The material shall be protected from light and stored in a cool and dry place.

4.3 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the Standard Mark may be granted to manufacturers or processors, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

6. TESTS

6.1 Tests shall be conducted as prescribed in col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *item* (ii)]

DETERMINATION OF MELTING RANGE

A-0. GENERAL

A-0.1 Outline of the Method — The melting range of a material is the range between the corrected temperature at which the material begins to form droplets and the corrected temperature at which it completely melts, as shown by the formation of a meniscus.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

†Specification for water for general laboratory use (*second revision*).

A-1. APPARATUS

A-**1.1 Bath** — a glass container, such as a small beaker or a Thiele tube provided with means of stirring and of controlled heating, is filled to the proper level with light paraffin oil, liquid silicone, or any other suitable fluid. The entire apparatus shall be well shielded from draught.

A-1.2 Thermometer — range from −20° to +150°C, 76-mm immersion and reading to 0·1°C accuracy. It shall further be of the solid stem, mercury-in-glass type with a cylindrical bulb.

A-1.3 Capillary Tube — 9 to 10 cm long, 1·5 to 2·0 mm external diameter, 0·2 to 0·3 mm wall thickness with one end closed.

A-2. PROCEDURE

A-2.1 Grind the sample to a fine powder. Dry the sample and empty the capillary tube by storing for 24 hours over sulphuric acid in a desiccator. Charge the capillary tube with sufficient powdered sample to form a closely packed column, about 2·5 mm high in the bottom of the tube, after the tube has been tapped against a solid surface.

A-2.1.1 Immerse the thermometer to its standard depth in the bath, making sure that the lowest part of the bulb is at least 2 cm above the bottom. Heat the bath to about 30° below the expected melting range. Attach the capillary tube to the thermometer by any suitable means so that the sample is level with the bulb. Re-immerse the thermometer and continue heating with constant stirring at a rate of 3° per minute to a temperature 30°C below the expected beginning of the melting range. Then carefully adjust the temperature rise to 1 to 2° per minute until the melting is complete.

A-2.1.2 Note the temperature at which the sample collapses against the side of the tube (beginning melting point) and the temperature at which the sample is liquid throughout (end melting point). Both of these temperatures shall fall within the specified melting range.

APPENDIX B

[*Table* 1, *item* (iii)]

ALKALI STABILITY TEST

B-0. GENERAL

B-0.1 Outline of the Method — Due to the presence of impurities, the material discolours when heated with alkali solution.

B-1. APPARATUS

B-1.1 Conical Flask — 100-ml capacity.

B-1.2 Water Bath

B-2. REAGENTS

B-2.1 Sodium Hydroxide Solution — 1 N, approximately.

B-3. PROCEDURE

B-3.1 Place 1 g of the sample with 25-ml of sodium hydroxide solution in a conical flask. Heat the mixture over a water-bath for 30 minutes and observe its colour.

B-3.1.1 The material shall be taken to have passed the test if a dark brown colour is not produced. Yellow colour, if developed, shall be disregarded for the purpose of this test.

NOTE — Exposure to strong sunlight causes a discoloration of the artificial musks and this should not be confused with discoloration due to impurities.

APPENDIX C

[*Table* 1, *item* (iv)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF MUSK KETONE

C-0. GENERAL

C-0.1 The chromatographic conditions given here are for guidance only.

C-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is

injected into the gas chromatograph when it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample uudergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

C-1. APPARATUS

C-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for musk ketone using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Sample	Musk ketone
a) *Column*:	
1) Material	Copper
2) Length	6 m
3) Outer diameter	0·64 cm
4) Inner diameter	0·48 cm
5) Stationary phase	Carbowax 20 M, 10 percent by mass
6) Solid support	Chromasorb WAW 60-80 mesh
b) *Carrier gas*	Nitrogen
c) *Conditions*:	
1) Column temperature isothermal	205°C
2) Injection port temperature	200°C
3) Carrier gas flow rate	50 ml/min
4) Inlet pressure	3·5 kg/cm^2
d) *Detector*:	
1) Type	Flame ionization detector
2) Temperature	280°C
e) *Recorder*:	
1) Span	1 mV
2) Chart speed	0·25 cm/min
f) *Attenuation*	64

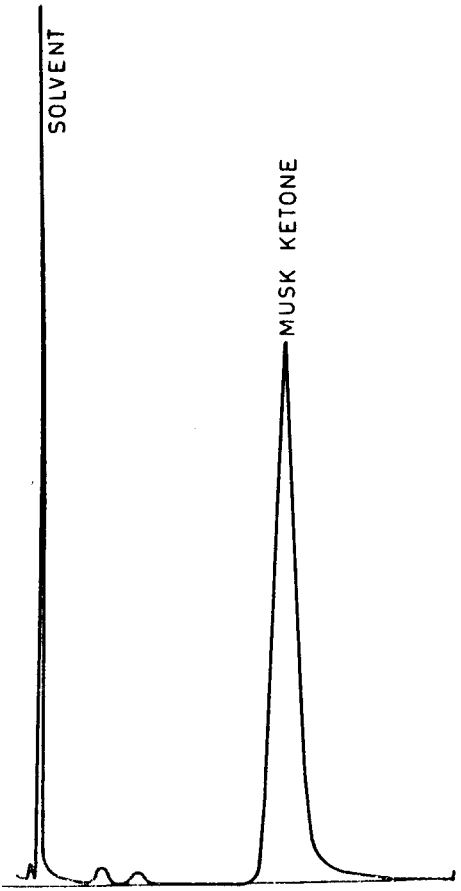


FIG. 1 TYPICAL CHROMATOGRAM OF MUSK KETONE

C-2. PROCEDURE

C-2.1 Conduct the flow of the carrier gas and inject the sample at inject port when it is vaporized and well mixed with the carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents

leaving the column. The detector signal, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents.

C-3. CALCULATION

C-3.1 Area Measurement (*see* Note 1) — Since normal peaks approximate a triangle the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

C-3.2 Area Normalization (*see* Note 2) — By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by totol area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is relative or indirect calibration.

(*Continued from page* 2)

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